PTO 04-4005

CY=JA DATE=19901009 KIND=A
PN=02-251607

CELLULOSE ESTER FIBER SPINNING METHOD
[Seruroosu esuteru sen'i no boushi houhou]

Toru Uenishi, et al.

UNITED STATES PATENT AND TRADEMARK OFFICE
Washington, D.C. June 2004

Translated by: FLS, Inc.

PUBLICATION COUNTRY	(19):	JP
DOCUMENT NUMBER	(11):	020251607
DOCUMENT KIND	(12):	A [PUBLISHED UNEXAMINED APPLICATION]
PUBLICATION DATE	(43):	19901009
APPLICATION NUMBER	(21):	01067157
APPLICATION DATE	(22):	19890317
INTERNATIONAL CLASSIFICATION	(51):	D 01 F 2/28; B 01 D 71/14; D 01 F 2/28
INVENTORS	(72):	UENISHI, TORU; MIYASHIRO, MORIO; MOMIYAMA, ZENJIRO
APPLICANT	(71):	TOYOBO CO., LTD.
TITLE	(54):	CELLULOSE ESTER FIBER SPINNING METHOD
FOREIGN TITLE	(54A):	SERUROOSU ESUTERU SEN'I NO BOUSHI HOUHOU

SPECIFICATION

1. Title of the Invention

CELLULOSE ESTER FIBER SPINNING METHOD

2. Claim

(1) With respect to spinning methods in which a solution obtained by dissolving cellulose ester in an organic solvent is pushed out of a nozzle, a cellulose ester fiber spinning method characterized by organic acid that contains two or more carboxyl groups and/or the salt of said organic acid being admixed to said solution.

3. Detailed Explanation of the Invention

(Field of Industrial Application)

The present invention pertains to spinning methods in which a solution obtained by dissolving cellulose ester in an organic solvent is pushed out of a nozzle, and its purpose is to supply a method that can suitably be applied to the manufacture of cellulose ester fibers that are applied for apparel, separation membranes, etc.

(Prior Art)

Cellulose ester fibers have conventionally been utilized for clothing, and are often utilized as separation membranes today. Their manufacture, namely spinning, has been carried out by means of dry or wet spinning, but both methods are plagued in terms of operation by broken threads. In order to solve this problem, the present invention introduces a thread breakage preventing method and supplies a cellulose ester fiber spinning method that is stable in terms of operation.

(Means for Solving the Problem)

The present invention was attained as a result of diligent study performed to solve the above problem. In other words, with respect to spinning methods in which a solution obtained by dissolving cellulose ester in an organic solvent is pushed out of a nozzle, the present invention is a cellulose ester fiber spinning method characterized by organic acid that contains two or more carboxyl groups and/or the salt of said organic acid being admixed to said solution.

In the present invention, the most desirable spinning mode for cellulose ester fibers is hollow fibers.

Various attempts have been made in the past to obtain methods for preventing threads from breaking during the cellulose ester fiber spinning process, such as changing the compositions of the source solutions, such as the polymer or solvent, and refining the raw materials.

However, an ultimate and effective means could not be discovered. The present inventors discovered that the cause of thread breakage originated in the small amount of metal ions contained in the raw-material cellulose ester. In other words, precipitation of the metal ions caused the formation of heterogeneous threads, which generated a physically adverse effect and caused the threads to break.

As a counteraction for this thread breakage, the removal of the metal ions from the cellulose ester is conceivable, but the separation is very difficult since they only exist in a very small amount. In light of this, an attempt was made to increase the operational stability by admixing

organic acid that contained two or more carboxyl groups and/or the salt of said organic acid with the cellulose ester solution and by thus generating a chelate complex and trapping the metal ions, which were the cause of thread breakage. As a result, thread breakage was significantly reduced or was eliminated, and the operability of the manufacture of cellulose ester fibers dramatically improved. Thus, the present invention was completed.

Next, the present invention will be explained in further detail.

The following are usable cellulose esters: derivatives of cellulose acetate, cellulose triacetate, etc.; and cellulose-mixed esterified substances such as cellulose acetate butyrate, cellulose acetate propionate, etc. When utilizing cellulose acetate, usually its degree of acetylation is mainly 30~65%, and when a different esterified derivative is utilized, usually the degree of its acylation is mainly 30~65%.

Representative examples of the above-described solvent in which cellulose ester is dissolved are the following: dimethylacetoamide, dimethylformamide, dimethylsulfoxide, tetramethylenesulfone, γ-butyrolactone, acetone, methylethylketone, methyl acetate, ethyl lactate, dioxane, tetrahydrofuran, methylenechloride, methylcellusolve, methylcellusolve acetate, nitromethane nitropropane, furukuraaru [as transliterated], glacial acetic acid, N-methyl-2-pyrollidone, etc.; or mixed solvents in which these solvents are the main ingredients, such as nitromethane-methanol, nitropropane-ethanol, nitropropane-methanol, methylenechloride-ethanol, methylenedichloride-ethanol, acetone-ethanol, etc.

Other than these, the following may be utilized instead: acetone-water; acetone-triethyleneglycol; acetone-methanol; acetone-benzol; etc.

As the organic acid of the present invention that contains two or more carboxyl groups, citric acid, oxalic acid, EDTA, etc. and their salts can be mentioned. It is desired that the carboxyl groups be in close proximity to one another inside the molecules.

As for the amount of this organic acid that is to be admixed, it needs to be an amount that is equivalent to 0.5~5 mol, more preferably 1.0~3.0 mol, in correspondence with the density of the very small amount of metal ions contained in the cellulose ester solution.

Next, a working example will be mentioned for a more detailed explanation.

(Working Example)

Cellulose triacetate was dissolved in N-methyl-2-pyrollidon and was made to be two types of source thread solutions by admixing citric acid to only one of them. By pushing these out from a double-tube nozzle, hollow fibers were made by means of wet and dry spinning methods. The results are indicated in Table 1.

Table 1

Source Solution Composition			Internal Solution Composition		Broken Thread Count (count/250km yarn)
cellulose triacetate (%)	N-methyl-2-pyrrolidone (%)	citric acid (ppm)	N-methyl-2-pyrrolidone (%)	H_2O (%)	
30.0	70.0	20 0	60.0	40.0	0 13

As is clear from the results of the broken thread counts of Table 1, the spinning properties improved remarkably by admixing citric acid to the source thread solution.

In addition, the performance of the hollow fibers obtained by spinning after admixing citric acid was exactly the same as the article without the citric acid, and citric acid, which forms a complex with metal ions, did not influence the performance and quality of the hollow fibers.

(Effects of the Invention)

As mentioned earlier, by admixing organic acid containing two or more carboxyl groups that are located in close proximity to one another to a raw spinning solution consisting of cellulose ester, the present invention supplies a cellulose ester fiber spinning method that allows the operability to be excellent with only few thread breakage occurrences.